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Procedia Engineering 10 (2011) 857–862
Engineering Procedia

ICM11

Compressive strength and splitting tensile strength of Zr-based metallic glass

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Abstract

A Compressive test on Zr-based metallic glass was performed using a universal testing machine. The fracture process was recorded by high-speed video camera with a recording rate of 125,000 fps. The specimen was shear-fractured at 1.9 GPa accompanied by the strong light emission. The fracture surface consisted of the brittle and vain-patterned surfaces. In addition, another compressive test was carried out in argon gas. Since light emission was not observed, it was evident that the light emission was a result of the oxidization of hot particles. In order to identify the beginning of the fracture, a splitting tensile test was carried out. Light emission began at the centre of the cross-section of a cylindrical specimen, and the crack was propagated in a direction of about forty-five degrees from the horizontal. The fracture criterion of Zr-based bulk metallic glass is not determined by the principal stress. The effect of the fraction of crystallization in Zr-based bulk metallic glass on light emission was also investigated using a specimen heat-treated at several temperatures. The relation between the fraction of crystallization and light emission was confirmed by the experimental analysis.

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Keywords : Zr-based metallic glass ; Light emission ; Fraction of crystallization ; Heat treatment

1. Introduction

A bulk metallic glass has many excellent properties (high strength, high corrosion resistance, et al.) because of its amorphous structure. Additionally, it also exhibits super-plasticity when super-cooled, so micro-machine components can be easily produced under very low stress. On the other hand, it has been reported that light emission was observed at the fracture of Zr-based metallic glass, and it is suggested that this light emission is the result of the oxidization of hot particles in air [1]. When the bulk metallic glass is used in the development of devices such as micro-geared motor, the light emission caused by the fracture of the bulk metallic glass component due to unexpected loading may induce serious damage through inflammability.

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Selection and peer-review under responsibility of ICM11 doi:10.1016/j.proeng.2011.04.141

A compressive test on Zr-based metallic glass was carried out to examine the relation between a fracture and light emission. However, since the initiation of the fracture and direction of the crack propagation cannot be controlled in the test, the correspondence between crack propagation and light emission was not identified, because the fracture surface was composed of several surfaces. Subsequently, a splitting tensile test to estimate the tensile strength of the brittle material was performed to clarify the relation between fracture morphology and light emission. Additionally, since the observation of the light emission under the dynamic tensile test [2] suggested that it was affected by the volume fraction of crystallization of the bulk metallic glass, this was also examined by the compressive test and splitting tensile test, and it became evident that the light emission was indeed dependent on the volume fraction of crystallization.

2. Materials and experimental methods

The material for the two loading tests was Zr-based metallic glass containing $Zr_{55}Al_{10}Ni_5Cu_{30}$. The diameter and length of the specimen for each test were 5.0 mm and 4.5 mm, respectively. The loading velocity was 1.0 mm/min. The super-cooling area was 750~800K under a heating rate of 5K/s. A high speed video with a maximum recording rate of 125,000 fps was used to record the brittle fracture of the specimen. The fracture surface was observed by laser microscope. The complete fracturing process was captured on optical camera film in the darkroom and the amount of light emitted at the fracture was calculated as the ratio of area measured from the binarized image. The specimen was heat-treated to change the volume fraction of crystallization at several temperatures. The structural changes in the heat-treated specimen were measured by X-ray diffraction (XRD) and differential scanning calorimetry (DSC). The X-ray target was Cu Kα and the scanning area was from 20 to 120 degrees. DSC measurement was performed to characterize the transformation behaviours from the amorphous structure in N_2 atmosphere at a heating rate of 30 K/min. Specimens for DSC measurement and X-ray diffraction were cut using an electro-spark machine to avoid heat generation.

3. Results and Discussions

3.1 Load-displacement curves and fracture process of compressive test and splitting tensile test

The load-displacement curves measured by the compressive test and the splitting tensile test at room temperature are shown in Fig.1. The longitudinal elastic modulus obtained by the compressive test is about 70 GPa, and the fracturing specimen was accompanied by the strong light emission at 1.9 GPa as shown in Fig.2.

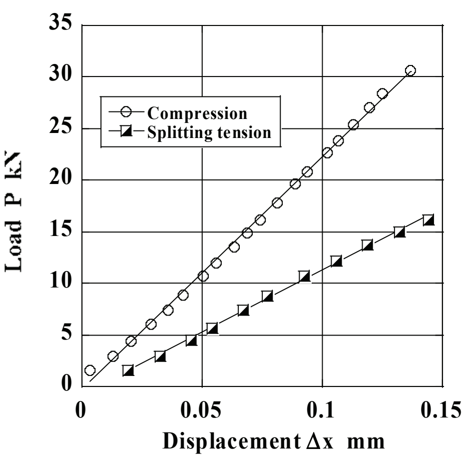


Fig. 1 Load-displacement curves

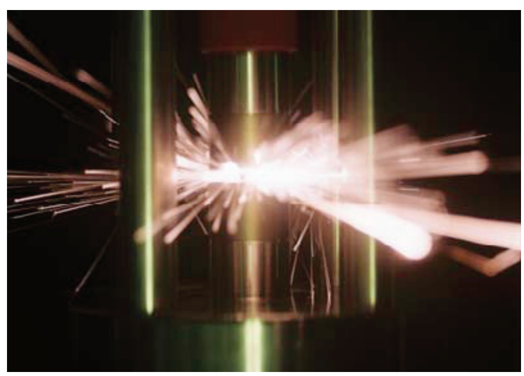
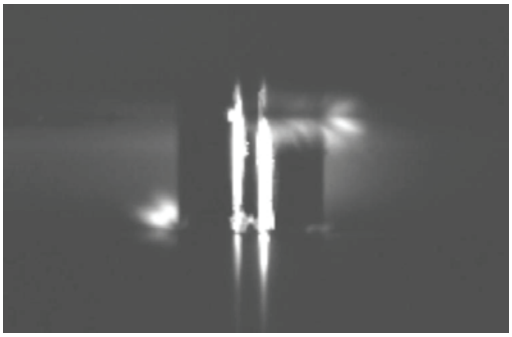
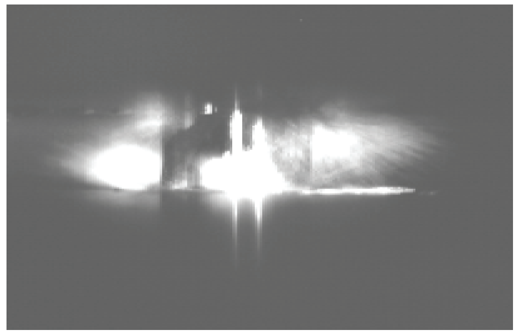


Fig. 2 Light emission from the compressing specimen

The initial and intermediate stages of the fracture of the compressing specimen are shown in Fig. 3 (a) and 3 (b). The two wide vertical lines in front of the specimen are reflections of the light source. Since the starting point of the fracture and propagating direction of the crack in the compressing specimen cannot be anticipated, it is not easy to sufficiently identify the fracture process by observation from a single viewpoint as in these camera photographs. As a result of the complex compressive fracturing process, the specimen was broken into several fragments. On the other hand, in the initiation and process of the fracture of the splitting tensile specimen (Fig. 4 (a) and (b)), the starting point of the fracture and propagating direction of the crack can be seen by the observing the circular cross-section of the specimen. In Fig. 4(a), the fracture is initiated at the centre of the specimen, and the crack is propagated in a direction of about 45 degrees to the loading direction, as seen in Fig. 4(b). The specimen loaded in the splitting tensile test was broken into just two pieces because its fracture morphology is simple.

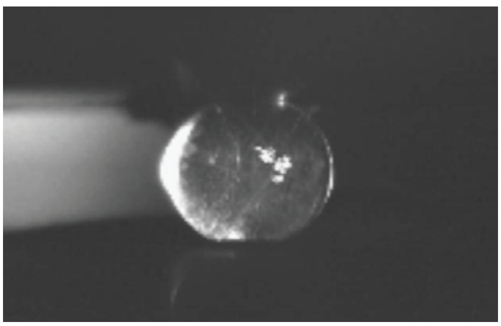


(a) Initial stage

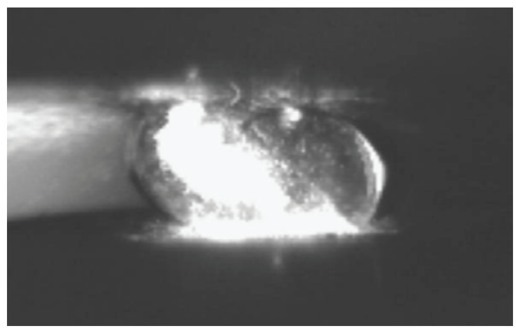


(b) Intermediate stage

Fig. 3 Fracture morphology of compressive test



(a) Initial stage



(b) Intermediate stage

Fig. 4 Fracture morphology of splitting tensile test

The splitting tensile test is usually used to estimate the tensile fracture stress of a brittle material like concrete. In a case of a brittle material, it is expected that the fracture surface is formed parallel to the loading direction because the fracture is controlled by the maximum principal stress. However, the fracture angle of Zr-based metallic glass generated by the splitting tensile test was at about 45 degrees to the loading direction, so the brittle fracture of bulk metallic glass is different from the fracture morphology of brittle materials like concrete. Yoshikawa et al. examined the yield surface (the fracture surface) of Zr-based metallic glass containing of $Zr_{60}Al_{10}Ni_5Cu_{25}$ and indicated that the yield surface is defined by the maximum shearing stress rule, called the yield condition of Tresca or Mohr-Coulomb rule [3]. Although the stress profile formed in the specimen by the splitting tensile test is not simple, the fracture morphology as seen in Fig. 4(b) suggests that the fracture of Zr-based metallic glass is controlled by the shear stress.

The relative amounts of transient light emission at each fracture stage induced by the compressive test and splitting tensile test are compared in Fig.5. The light emission from the compressive fracture indicates a simple increase, while that caused by the splitting tensile fracture indicates a two-step increase. In the splitting tensile fracture, when the main crack propagates in a direction of about 45 degrees from the loading direction (Fig.4 (b)), the fragments on the right radiate light first, and thereafter the fragments on the left radiate light. These results show that the light emission process of the splitting tensile fracture represents a two-step increase.

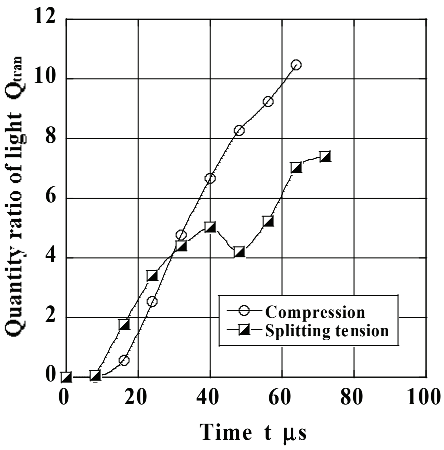


Fig.5 Transient light emission in compressive test and splitting tensile test

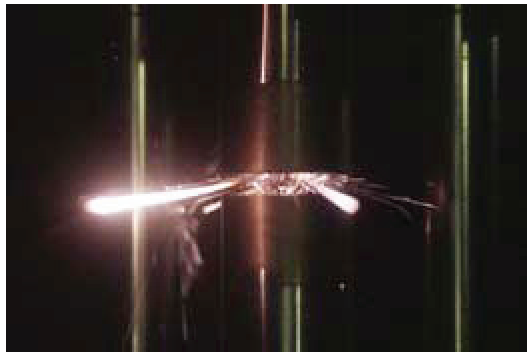


Fig.6 Light emission of a specimen heat-treated at a temperature of 700K

3.3 Effects of heat treatment on light emission

Sakino et al. have suggested that reduction of the volume fraction of crystallization may induce the decrease in light emission [2]. Since the volume fraction of crystallization is controlled by heat treatment, the specimen was heat-treated at several temperatures. The heating rate was 0.60 K/s and the holding time at each maximum temperature was 900 s. The light emission morphology of the specimen heat-treated at a temperature of 700K is shown in Fig.6.

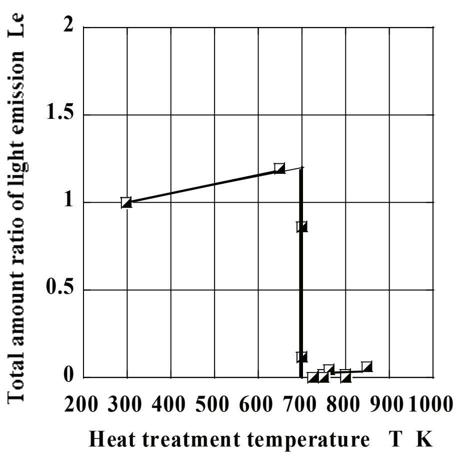


Fig.7 Effect of heat treatment on light emission

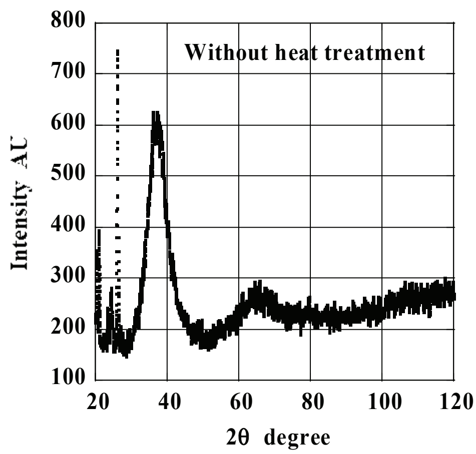


Fig.8 X-ray diffraction pattern

As shown in Fig.6, the light emission from the specimen heat-treated at a temperature of 700K is much less than that of the untreated specimen in Fig.2. It is evident from Fig.6 that light emission is controlled by heat treatment. The total amount of light emission recorded during the complete fracturing process is binarized and normarized using the amount of light emitted by the untreated specimen. The effect of heat treatment on light emission is shown in Fig.7. The amount of light emission simply increases up to 700K, and thereafter rapidly decreases to nearly zero near the same temperature. It is deduced that the increase up to 700K is caused by the transition from a several-fracture surface formation to a multi-fracture surface formation. As shown in Fig.7, the light emission then drastically decreases at 700K or more. In order to investigate the cause of the decrease, the change in the crystal structure of each heat-treated specimen was examined by XRD and DSC.

The X-ray diffraction pattern of the untreated specimen is shown in Fig.8. The broad maximum area characterizing the bulk metallic glass is seen between 30 and 50 degrees. The sharp peak at the 25 degree is from the compound gluing the specimen to the plate. In order to evaluate the effect of temperature on the XRD pattern, a half-width of 2θ indicating the width of the broad maximum area was used. As shown in Fig. 9, the half-width of the broad diffraction maximum of the untreated specimen shows about 6.6 degrees and the half-width gradually decreases up to 700K. However, the rate decreases drastically when the heat treatment temperature just exceeds 700K and the half-width of the broad maximum reduces down to about 4.5 degrees. The effect of heat treatment temperature on heat flow per unit mass measured by DSC is shown in Fig.10. The transformation from amorphous to crystal structure does not occur below 700K, and the heat flow above 700K is nearly zero. It is clear from the results of the XRD and DSC that the drastic decrease in the light emission at 700K is caused by the structural change in the Zr-based metallic glass. Since the glass transition temperature and melting temperature of $Zr_{55}Al_{10}Ni_5Cu_{30}$ are 681K and 777K, respectively, it is deduced that light emission is controlled by glass transition temperature.

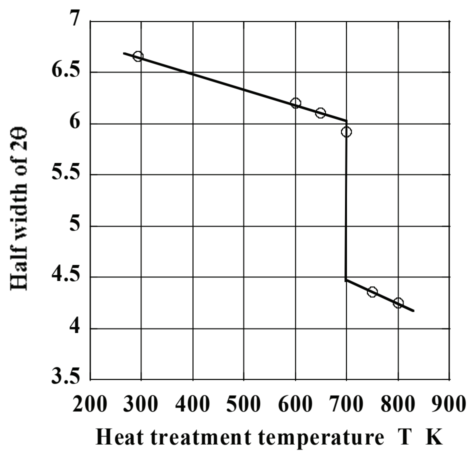


Fig.9 Effect of heat treatment on half-with of broad maximum area

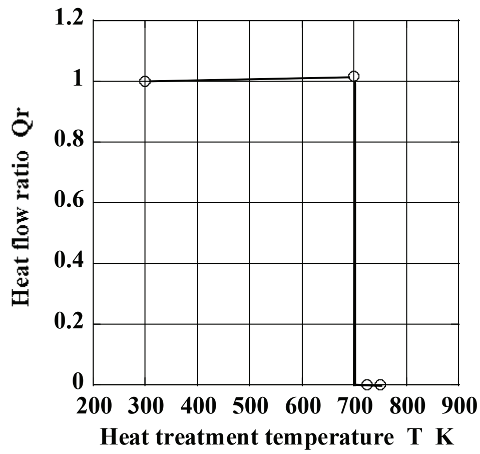


Fig.10 Effect of heat treatment on the heat flow

3.4 Comparison of fracture surface generated by compression and splitting tension

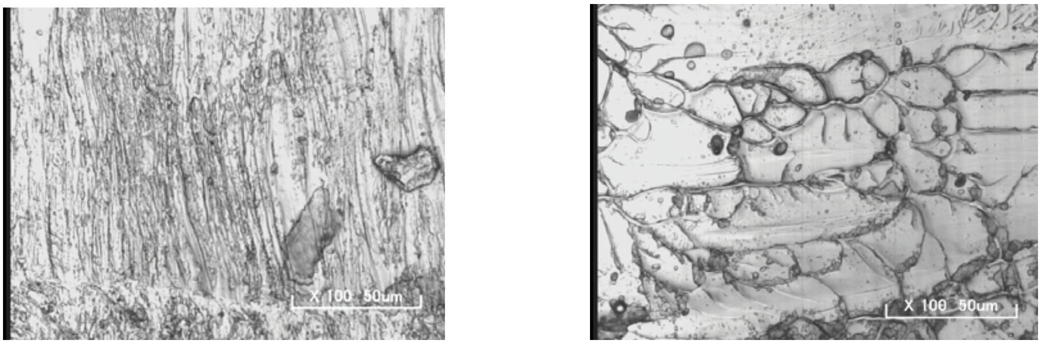
As described above, the fracture morphologies generated by the compressive test and splitting tensile test are different in that the compressed specimen was divided into several pieces by the fracture while the splitting tensile specimen was divided into two pieces. When the fracture surface is a single surface, it is important to examine the fracture angle relative to the loading direction [4]. However, when the fracture surface is composed of several surfaces, it is also important to evaluate the fracture surface morphology. In the case of the compressive test, the area near the compressing face deduced to be the starting point of the fracture is a brittle face, as shown in Fig.11 (a). On the other hand, other fracture faces presenting a veined pattern indicate a high-temperature fracture as shown in Fig.11 (b). The fracture surface generated by the splitting tensile test is shown in Fig.12. The whole of the fracture surface was covered by a pattern of large and small veins, as shown in Fig12. A compressive test was also performed in argon gas. Although light emission was not confirmed by visual observation, a veined pattern was observed on the fracture surface as seen in Fig.13, and it is deduced that the light emission is caused by the oxidization of hot particles.

4. Conclusions

The compressive test and splitting tensile test of Zr-based bulk metallic glass, $Zr_{55}Al_{10}Ni_5Cu_{30}$, were conducted and the following results are obtained.

1) The fracturing of Zr based bulk metallic glass is accompanied by the strong light emission in compressive test and the splitting tensile test

2) The fracture morphology generated by the compressive test is complex, while the fracture morphology generated by the splitting tensile test is simple.
3) Light emission is controlled by heat treatment, and the amount of emitted light is dependent on the volume fraction of crystallization.
4) Light emission is caused by the oxidization of hot particles.



(a) Starting point area of fracture (b) Vein-patterned area

Fig.11 Fracture surface morphology generated by compressive test

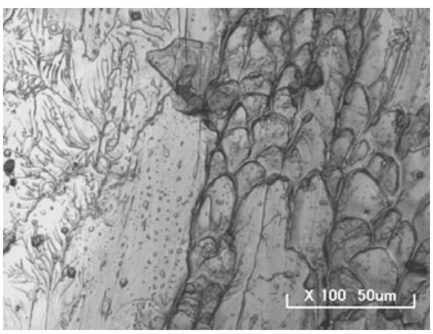


Fig.12 Fracture surface morphology generated by splitting tensile test

Fig.13 Fracture surface morphology generated by compressive test in argon gas

Acknowledgement

The authors are grateful to Visiting Prof. Xinmin Wang, Institute for Materials Research, Tohoku University, for providing the specimens. The authors express my appreciation to Mr. Horiuchi, an undergraduate student in Tohoku Gakuin University, for his assistance in the experiment.

References

[1] Gilbert C. J., Ager III J.W., Schroeder V., Ritchie R. O., Light emission during fracture of a Zr-Ti-Cu-Be bulk metallic glass, APPLIED PHYSICS LETTERS 1999;74:3809-3811.

[2]Sakino Y., Kuroda T., Kobayashi A., Y.-C. KIM, Fracture behaviours of $Zr_{48}Cu_{36}Al_8Ag_8$ and $Zr_{55}Cu_{30}Al_{10}Cu_{30}$ bulk metallic glasses in high speed tensile test, Frontier of Applied Plasma Technology 2010; 3:102-106.

[3] Yoshikawa T., Maeoka Y., Inaba T., Tokuda M., Simple shear test of Zirconium-based bulk metallic glass and yield surface at room temperature, J. of the Society of Materials Science, Japan 2010; 59:110-117.

[4] Zhang Z. F., Eckert J., Schultz L., Difference in compressive and tensile fracture mechanisms of $Zr_{59}Cu_{20}Al_{10}Ni_8Ti_3$ bulk metallic glass, Acta Materialia 2003; 51:1167-1179.